A Study of the Applicability of Atomic Emission Spectroscopy (AES), Fourier Transform Infrared (FT-IR) Spectroscopy, Direct Reading and Analytical Ferrography on High Performance Aircraft Engine Lubricating Oils

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Abstract: The Joint Oil Analysis Program Technical Support Center (JOAP-TSC) was tasked by the U.S. Air Force to determine the appropriateness of various oil analysis techniques to provide early warning of abnormal operating conditions. The study analyzed ~1300 samples from F-16, F110-GE100 gas turbine engines by spectroscopy (AES and FT-IR) and direct reading and analytical ferrography. A statistical analysis of the data collected is presented.

Key Words: Analytical ferrography; atomic emission spectroscopy; condition monitoring; direct reading ferrography; Fourier transform infrared spectroscopy; FT-IR; JOAP; JOAP-TSC; oil analysis.

Introduction: The U.S. Air Force Oil Analysis Program (AFOAP) uses atomic emission spectroscopy for determination of equipment wear. This study was initiated to determine whether or not there were any aircraft engine failure modes that *only generate wear metals too large to be measurable by AES.* In addition, oil failure modes such as contamination and degradation were monitored to determine any potential correlation with mechanical failure modes. With the exception of the Complete Oil Breakdown Rate Analyzer (COBRA) [1 & 2] test for PW-F100 engines, the AFOAP does not monitor oil condition. The General Electric F110-GE100 engine was chosen by the Air Force for this study.

Test Equipment and Methods: The JOAP-TSC analyzed F110 engine samples (from Pope AFB and Cannon AFB) by atomic emission spectrometry (AES), analytical ferrography, direct reading ferrography (DRIII) and Fourier transform infrared (FT-IR) spectroscopy. A database was created using pertinent squadron information obtained from the DDF 2026, sample submission form. To speed sample and statistical analyses, the AES, DRIII and FT-IR were linked via a LAN system for automatic data entry into the database. Manual entry was required only for the analytical ferrography data.

The following approach was taken for sample analysis.

a. The Air Force analyzed all samples by AES and forwarded them to the JOAP-TSC.

b. The JOAP-TSC analyzed all samples by AES (Baird MOA) and FT-IR (Bio-Rad "Oil Analyzer"). The JOAP-TSC AES results were similar to the Air Force's AES results.

c. Approximately every fifth sample was analyzed by DRIII and analytical ferrography (Predict/DLI). This approach was taken due to the amount of time required for analytical ferrography and the small sample volume—20 ml bottles—generally with only 5 to 10 mls remaining. If the fifth sample did not have sufficient oil, the fourth or sixth sample was chosen, if volume permitted. In the case of abnormal results from any test (AES, FT-IR or ferrography) and volume permitting, the intermediate samples were analyzed by analytical ferrography. The tests were generally performed in the following order: DRIII, ferrrography, FT-IR and AES. Note: AES was performed at field laboratories on all samples.

d. AES analysis guidelines were taken from T.O. 33-1-37-3 [3].

e. Fluid condition/contamination levels for FT-IR were based on preliminary studies of over 1000 polyol ester lubricant (Mil-L-7808) samples [4].

f. No statistical guidelines or limits were available for the ferrographic analysis of oil in F110-GE100 engines.

Results Analysis: The study evaluated over 1300 samples taken from 48 engines (28 aircraft). Engines removed for phase often returned to the study in another aircraft. The results were compared to available limits and trended over time. The trend calculations required multiple samples (same engine) consequently, the number of samples for which trends were calculated is lower (686). Scheduled engine removals occurred throughout the study further limiting the number of samples from any single engine.

The sample data was used to calculate limits based on average and standard deviation data. The AES calculated limits were compared to the engine T.O. limits to examine limits behavior. Differences between predicted and T.O. limits are discussed under exceptions. Example data is shown as trend plots along with explanations. Please note, there were no high wear or severe fluid condition or contamination indicators observed during the project. Field laboratories reported no engine failures had occurred.

Ferrography/Particulate Analysis: The severity of wear for the F110 engine was established by empirical means—subjective operator experience [5]. The severity of wear for each of the named wear modes is characterized by the index 0 (none) to 4 (severe). The index is specific to this engine and this study.

The most commonly seen particles were created by normal rubbing wear and bearing wear. Neither of these wear modes were seen in critical amounts. Some black oxides, red oxides, sand and fibers were also seen in trace amounts. Molybdenum disulfide, used as an anti-seize compound on fasteners, was also common. The majority of sample slides were clean—no particulate debris. Since there were no wear related failure modes, the DRIII results were nominal.

Particle Exceptions: There were a few instances of metal particles larger than 100 microns when AES and FT-IR were normal. The large particles were not present in subsequent samples and are not believed to have been related to any failure mode. Table I shows an example. Table II contains the statistics and limits for all ferrography data.

Sample ID	Date	CHUNKS NF	CHUNKS NF	LAMINAR	LAMINAR S	LAMINAR
96S00169	Apr27/96	0.00	0.00	2.00	20.00	0.0
96S00170	May2/96					
96S00171	May2/96					
96S00172	May2/96	0.00	0.00	0.00	0.00	1.0
96S00173	May6/96					
96S00174	May7/96	1.00	140.00	1.00	10.00	1.0
96S00221	May7/96					
96S00294	May7/96	0.00	0.00	1.00	16.00	0.0
96S00295	May8/96	0.00	0.00	1.00	0.00	0.0
96S00296	May13/96					
96S00343	May14/96	0.00	0.00	0.00	0.00	0.0
96S00344	May14/96					
96S00345	May15/96					
96S00368	May21/96	0.00	0.00	1.00	18.00	0.0
96S00369	May21/96					
96S00370	May29/96	1.00	40.00	0.00	0.00	2.0

Table 1: Example of Wear Particle Results

Atomic Emission: All AES results for most samples were within limits. An occasional sample indicated an elevated level or trend, primarily for molybdenum and zinc. Generally, subsequent samples showed normal readings (Table III). Spurious data such as this raises suspicions of contaminated samples or poor tests. Molybdenum disulfide is used as an antiseize compound on fasteners and can potentially enter the oil. Zinc is a common component of additives in ground vehicle lubricants and is also found in copper alloys and galvanized steel. The source of the zinc in these cases is unknown.

Sample Date	PPM Zn
15 Jan	1
16 Jan	14
16 Jan	0

Table III. Example of Zinc Data (all samples analyzed on the same day)

Table II: DRII and Analytical Ferrography Statistics and Limits

Parameter		Number of					Calculated Limits		Existing Limits	
Name	Type	Samples	Average	Std Dev	Min	Max*	2 Std Dev	4 Std Dev	Marginal	High
Blk Oxides	level	274	n/a	n/a	0	n/a	n/a	n/a	none	none
Red Oxides	level	274	n/a	n/a	0	n/a	n/a	n/a	none	none
Rubbing Wear	level	274	n/a	n/a	0	n/a	n/a	n/a	none	none
Fibers	level	274	n/a	n/a	0	n/a	n/a	n/a	none	none
Fiction Polymers	level	274	n/a	n/a	0	n/a	n/a	n/a	none	none
Mo Disulfide	level	274	n/a	n/a	0	n/a	n/a	n/a	none	none
Corrosive Wear	level	274	n/a	n/a	0	n/a	n/a	n/a	none	none
Sand/Dirt	level	274	n/a	n/a	0	n/a	n/a	n/a	none	none
Chunks Ferr	level	274	n/a	n/a	0	n/a	n/a	n/a	none	none
	size	274	3.34	n/a	0	200	n/a	n/a	none	none
Chunks Non-Fe	level	274	n/a	n/a	0	n/a	n/a	n/a	none	none
	size	274	8.86	n/a	0	200	n/a	n/a	none	none
Cutting Wear Fe	level	274	n/a	n/a	0	n/a	n/a	n/a	none	none
	size	274	0.78	n/a	0	40	n/a	n/a	none	none
Cutting Wear Non-Fe	level	274	n/a	n/a	0	n/a	n/a	n/a	none	none
	size	274	0.93	n/a	0	44	n/a	n/a	none	none
Laminar Fe	level	274	n/a	n/a	0	n/a	n/a	n/a	none	none
	size	274	2.69	n/a	0	50	n/a	n/a	none	none
Laminar Non-Fe	level	274	n/a	n/a	0	n/a	n/a	n/a	none	none
	size	274	4.52	n/a	0	180	n/a	n/a	none	none
Sliding Fe	level	274	n/a	n/a	0	n/a	n/a	n/a	none	none
	size	274	3.49	n/a	0	130	n/a	n/a	none	none
Sliding Non-Fe	level	274	n/a	n/a	0	n/a	n/a	n/a	none	none
	size	274	0.38	n/a	0	40	n/a	n/a	none	none
Spheres	level	274	n/a	n/a	0	n/a	n/a	n/a	none	none
	size	274	0.25	n/a	0	6	n/a	n/a	none	none
DRIII Large Part	level	286	2.19	1.37	0.00	11.60	4.93	7.67	none	none
DRIII Small Part	level	286	1.36	0.83	0.00	5.80	3.02	4.68	none	none
DRIII PLP	percent	286	22.59	22.67	0.00	100.00	n/a	n/a	none	none
DRIII WPC	level	286	3.52	1.92	0.30	14.10	7.36	11.20	none	none

Size values are in microns

*Note: Maximum value could not be determined due to lack of failure data

The predicted limits from the study are well below the T.O. limits. This difference is due to the very clean sample set, i.e., there are no failure modes represented. Table IV contains the statistics and limits for all atomic emission data.

Lubricant Condition (FT-IR): The severity of oil condition indicators were based on prior work with Air Force, Army and Navy aircraft FT-IR analysis. Most samples were normal—no indication of serious lubricant contamination or degradation. However, a change (loss) in antiwear additive level was noticed between makeup oil additions. If oil additions were made frequently (small quantities—one half to one pint), the change in antiwear levels was nominal. When large quantities of make-up oil were added (2 to 3 pints)—indicative of longer engine runtimes, the corresponding drop in antiwear additive was significant (Figure 1). The impact of the decrease in antiwear additives on engine reliability is beyond the scope of this study.

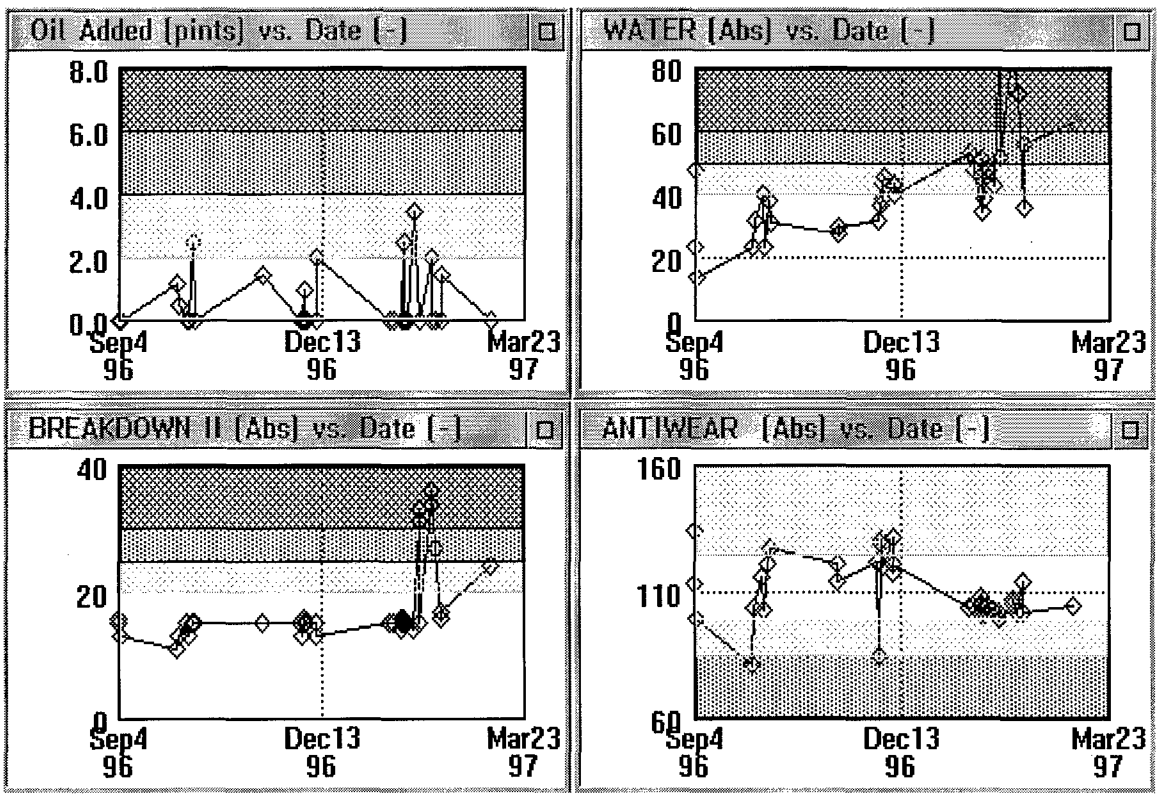

Figure 1. Example of Data Trends (Note: Out of limit areas are shaded.)

The study also indicated water contamination in excess of current alarm levels (45 = 1000 PPM). In addition, a few significant water contamination problems (water greater than 2000 PPM) were noted. High levels of water were always associated with some lubricant degradation (Figure 1). Water contamination is a known cause for polyol ester lubricant breakdown. Engine operating conditions (high temperatures) in conjunction with oil

Table IV: AES Statistics and Limits

Parameter		Number of					Calculated Limits		Existing Limits	
Name	Type	Samples	Average	Std Dev	Min	Max	2SD+Ave	4SD+Ave	Marginal	High
Ag	level	1321	0.01	0.12	0.0	3.0	0.25	0.49		
	trend	686	0.00	0.12	n/a	1.2	0.23	0.46	n/a	2
Al	level	1321	0.06	0.28	0.0	4.5	0.64	1.22	4	7
	trend	686	0.03	0.29	n/a	3.2	0.60	1.18	n/a	2
B	level	1321	0.03	0.11	0.0	2.0	0.25	0.47	none	none
	trend	686	-0.01	0.12	n/a	0.8	0.23	0.47	none	none
Ba	level	1321	0.01	0.12	0.0	2.0	0.25	0.49	none	none
	trend	686	-0.01	0.12	n/a	0.8	0.23	0.47	none	none
Cd	level	1321	0.16	0.41	0.0	4.0	0.98	1.80	none	none
	trend	686	0.00	0.36	n/a	2.1	0.72	1.44	none	none
Cr	level	1321	0.02	0.12	0.0	1.2	0.26	0.51	7	10
	trend	686	0.01	0.11	n/a	1.1	0.23	0.46	n/a	2
Cu	level	1321	0.76	0.64	0.0	5.0	2.05	3.34	13	20
	trend	686	-0.01	0.32	n/a	1.7	0.64	1.28	n/a	4
Fe	level	1321	0.09	0.34	0.0	8.3	0.76	1.43	9	14
	trend	686	0.00	0.19	n/a	1.0	0.39	0.78	n/a	3
Mg	level	1321	0.43	0.45	0.0	4.0	1.33	2.22	7	10
	trend	686	-0.01	0.39	n/a	2.9	0.77	1.54	n/a	2
Mn	level	1321	0.21	1.02	0.0	7.4	2.25	4.29	none	none
	trend	686	0.17	0.92	n/a	7.0	2.01	3.85	none	none
Mo	level	1321	1.09	1.02	0.0	11.0	3.13	5.16	3	5
	trend	686	0.12	0.86	n/a	10.5	1.84	3.56	n/a	2
Na	level	1321	5.71	2.60	0.0	18.7	10.91	16.11	none	none
	trend	686	0.12	2.24	n/a	12.7	4.60	9.08	none	none
Ni	level	1321	0.03	0.15	0.0	1.4	0.34	0.64	6	9
	trend	686	-0.02	0.18	n/a	1.0	0.35	0.71	n/a	2
Pb	level	1321	0.21	1.06	0.0	9.4	2.33	4.45	none	none
	trend	686	0.04	0.97	n/a	6.5	1.98	3.92	none	none
Si	level	1321	0.78	1.64	0.0	22.8	4.07	7.36	13	20
	trend	686	0.02	1.63	n/a	18.3	3.28	6.54	n/a	5
Sn	level	1321	5.53	1.52	0.0	15.3	8.57	11.61	none	none
	trend	686	-0.02	1.63	n/a	9.5	3.24	6.50	none	none
Ti	level	1321	0.14	0.29	0.0	3.3	0.72	1.31	6	9
	trend	686	-0.03	0.29	n/a	1.9	0.55	1.12	n/a	2
V	level	1321	0.77	1.52	0.0	28.2	3.81	6.85	none	none
	trend	686	0.07	0.76	n/a	10.3	1.59	3.11	none	none
Zn	level	1321	0.79	2.45	0.0	35.5	5.70	10.61	3	5
	trend	685	-0.07	2.66	n/a	16.2	5.26	10.58	n/a	2

Table V: FT-IR Statistics and Limits

Parameter		Number of					Calculated Limits		Existing Limits	
Name	**Type**	**Samples**	**Average**	**Std Dev**	**Min**	**Max**	**2 Std Dev**	**4 Std Dev**	**Marginal**	**Abnormal**
Water	level	1348	36.42	15.06	8.0	95.0	66.53	96.64	n/a	45
Breakdown I	level	1348	24.04	3.71	12.0	38.0	31.45	38.87	n/a	65
Breakdown II	level	1348	15.72	3.66	11.0	37.0	23.05	30.37	n/a	25
Antiwear*	level	1348	110.99	10.82	80.0	147.0	89.35	67.71	n/a	80
Fuel Dilution	level	1348	189.32	2.10	184.0	195.0	193.52	197.73	n/a	201
Other Fluid	level	1348	12.97	8.08	0.0	43.0	29.14	45.30	n/a	30

FT-IR values and limits are in Absorbance units
Note: water 45 Abs = 1000 PPM; breakdown I and II are for TAN of ~1.5 mg of KOH/ml
*Note: antiwear limits are calculated below mean

additions corrected both the water and degradation problems. Consequently for aircraft with frequent oil additions, water contamination levels should only be a concern if present during extended periods of non-operation. Table V contains the statistics and limits for all FT-IR data.

Conclusions: There was no data to corroborate the hypothesis of the study—that large wear particles are generated in the absence of smaller wear particles measurable by AES. In retrospect, there were insufficient samples to ensure that no relationship exists. In addition, no oil related engine failure modes were observed during the study and no engines were replaced because of mechanical defect or failure.

AES appears to generate reliable results for aircraft engine wear analysis. DRIII results were unreliable due to the low level of normal wear debris—very clean oil. *In the absence of engine failures,* the behavior of the DRIII readings and ferrography on samples indicating severe wear could not be determined.

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References:

1. Smith, H.A., "Complete Oil Breakdown Rate Analyzer (COBRA) for Identifying Abnormal Operating Turbine Engines", Proceedings JOAP International Oil Analysis Workshop, 1983.
2. Toms, A. M., C. Rizzo, G. R. Humphrey, A. Lang, "A Study on Instrumentation Techniques Available for the Early Detection of "Burnt Oil" in F100-PW-100/200/220/229 Engines, Part II", JOAP-TSC-TR-97-01, 18 Feb 1997.
3. Joint Oil Analysis Program Manual, T.O. 33-1037-3/T.M. 38-301-3/NAVAIR 17-15-50.3.
4. Toms, Allison M., "Bio-Rad FTS7 Fourier Transform Infrared (FT-IR), JOAP-TSC-TR-95-01 Final Report", 23 November 1994
5. Wear Particle Atlas, ed. D. P. Anderson, Predict Technologies, Report NAEC-92-163, June 1982